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RECOMMENDED SPECIFICATION FOR COMPOSITE THINNER FOR THINNING SEMIPASTE PAINTS WHEN THE USE OF STRAIGHT LINSEED OIL IS NOT JUSTIFIED

PREPARED AND RECOMMENDED BY THE U. S. INTERDEPARTMENTAL COMMITTEE ON PAINT SPECIFICATION STANDARDIZATION, SEPTEMBER 27, 1920; P. H. WALKER, BUREAU OF STANDARDS, CHAIRMAN; J. W. GINDER, TREASURY DEPARTMENT, SECRETARY

[This committee was appointed at the suggestion of the Secretary of Commerce, and consisted of representatives of the War, Navy, Agriculture, Interior, Post Office, Treasury, and Commerce Departments, the Panama Canal, and the Educational Bureau of the Paint Manufacturers' Association of the United States. The committee submitted a preliminary draft of the specification to a large number of representatives of the paint and varnish manufacturers, and gave careful consideration to the replies received.]

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1. GENERAL

This specification covers a composite thinner which contains in one liquid drying oil, drier, and volatile thinner. Such preparations are sometimes called "Thinning Mixtures for Paint," and are also offered under a variety of trade names, such as "Japan Oil," "Paint Oil," "Linseed Oil Substitute," etc. The last name should, however, not be used, for while such materials may have decided merit they are not substitutes for linseed oil.

The composite thinner must meet the following requirements:

APPEARANCE.—Shall be clear and free from suspended matter and sediment.

Color.—No darker than a solution of 6 g of potassium dichromate in 100 cc pure sulphuric acid of specific gravity 1.84.

Odor.—Not offensive, either in bulk or in its subsequent use in paint mixtures.

Mixing with Linseed Oil.—When mixed in any proportion with pure raw linseed oil meeting the specifications of B. S. Circular 82, the resulting mixture shall be clear and shall show no separation or precipitation on standing 18 hours.

Drying.—When flowed on glass, the composite thinner shall set to touch in not more than 4 hours and dry hard in not more than 6 hours. When mixed with an equal volume of pure raw linseed oil, the resulting mixture when flowed on glass shall set to touch in not more than 6 hours and dry hard in not more than 8 hours.

Toughness.—The film on glass after baking for 6 hours at 105 to 110° C (221 to 230° F) shall be glossy, tough, and elastic.

Nonvolatile Matter.—Not less than 50 per cent by weight.

Acid Number.—Not more than 12, calculated to basis of nonvolatile matter.

2. SAMPLING

It is mutually agreed by buyer and seller that a single package out of each lot of not more than 1000 packages be taken as representative of the whole. Whenever possible an original unopened container shall be sent to the laboratory, and when for any reason this is not done, the inspector shall thoroughly mix the contents of the containers sampled, transfer not less than 1 quart to a clean, dry, glass bottle or tin can, which must be nearly filled with the sample, securely stoppered with a new, clean cork or well-fitting cover or cap, sealed, and distinctly labeled by the inspector. The inspector should take a duplicate from the container sampled to hold for check in case of dispute, and when requested should take a sample for the seller.

3. LABORATORY EXAMINATION

Samples will, in general, be tested by the following methods, but the purchaser reserves the right to apply any additional tests or use any available information to ascertain whether the material meets the specification.

(*a*) Appearance.—Fill two test tubes of the same size (15 cm or 6 inches), with the thoroughly mixed sample to within 2.5 cm (1 inch) of the top. Stopper the tubes with clean corks. Let stand for 24 hours. Note whether sediment is evident in

the tubes; if not, shake one tube vigorously and compare the two tubes. If they still look alike and the liquid appears clear, the sample is considered free from sediment and suspended matter.

(*b*) COLOR.—Prepare a standard color solution by dissolving 6 g of pure powdered potassium dichromate in 100 cc of pure concentrated sulphuric acid of specific gravity 1.84. Gentle heat may be used if necessary to perfect the solution of the dichromate. The standard color solution and a sample of composite thinner to be tested shall be placed in clear, thin-walled glass tubes of the same diameter. The color comparison shall be made by placing the tubes close together and looking through them by transmitted light. The tubes used for this test should be 1.5 to 2.0 cm ($\frac{5}{8}$ to $\frac{13}{16}$ inch) in diameter and shall be filled to a depth of at least 2.5 cm (1 inch).

(Since the potassium dichromate-sulphuric acid must be freshly made for this color comparison, it is frequently more convenient to compare samples with a permanently sealed tube of composite thinner which has previously been found to be slightly lighter in color than the standard solution of 6 g dichromate in sulphuric acid. When samples are found to be darker than this sealed tube of composite thinner, the dichromate standard should be made up for final decision.)

(*c*) ODOR.—Note the odor of the material in bulk, pour a small portion in a shallow flat-bottomed dish, and allow to stand exposed to the air for not less than 48 hours. Flow some on a glass plate and allow to dry in a vertical position for not less than 48 hours. Note the odor of these test portions from time to time. A mild odor of wood turpentine or of fish oil should not be cause for rejection, but a pronounced offensive odor, either due to very crude wood turpentine, rank fish oil, or other offensive substances, would be cause for rejection.

(*d*) MIXING WITH LINSEED OIL.—Thoroughly mix 25 cc of the sample with 25 cc pure raw linseed oil and transfer portions of the mixture to two similar test tubes which shall be filled to within 2.5 cm (1 inch) of the top and stoppered with clean corks.

Thoroughly mix 5 cc of the sample with 45 cc of pure raw linseed oil and transfer portions of the mixture to two similar test tubes which shall be filled to within 2.5 cm (1 inch) of the top and stoppered with clean corks.

Let the four tubes stand for 24 hours and note if any sediment or curdling is apparent in any of the tubes. If not, shake one tube

of each pair vigorously and then compare with the unshaken tube of the same dilution. If the tubes of the same dilution still look alike, the sample is considered to mix properly with linseed oil.

(*e*) DRYING.—Pour the undiluted sample and the mixture with an equal volume of linseed oil on clean glass plates not less than 15 cm (6 inches) long and 10 cm (4 inches) wide. Place the plates in a nearly vertical position in a well-ventilated room but not in the direct rays of the sun. The temperature of the room should be from 21 to 32° C (70 to 90° F). The films are tested at points not less than 2.5 cm (1 inch) from the edges of the films by touching with the finger. The material is considered to have set to touch when gentle pressure of the finger shows a tacky condition but none of the material adheres to the finger. The material is considered to have dried hard when the pressure that can be exerted between the thumb and finger does not move the film nor leave a mark that remains noticeable after the spot is lightly polished.

(*f*) TOUGHNESS.—Pour the undiluted sample on a clean glass plate. Let drain in a vertical position for 2 minutes, then place in a horizontal position, film up, and let stand at room temperature for 2 hours. Then bake at a temperature of 105 to 110° C (221 to 230° F) for 3 hours. Remove from the oven and let stand at room temperature for not less than 18 hours. The resulting film shall be glossy and when tested with a knife blade shall show elastic properties, turning up with the blade of the knife in an elastic ribbon without cracking or breaking. The film shall also stand rapid light rubbing without breaking the surface.

(*g*) NONVOLATILE MATTER.—Place a portion of the sample in a stoppered bottle or weighing pipette. Weigh the container and sample. Transfer about 1.5 g of the sample to a weighed flat-bottomed metal dish about 8 cm in diameter (a friction-top can plug). Weigh the container again and by difference calculate the exact weight of the portion of sample transferred to the weighed dish. Heat the dish and contents in an oven maintained at 105 to 110° C (221 to 230° F) for 3 hours. Cool and weigh. From the weight of the residue left in the dish and weight of the sample taken calculate the percentage of nonvolatile matter.

(*h*) ACID NUMBER.—Place a portion of the sample in a stoppered bottle or weighing pipette. Weigh the container and sample. Transfer an amount corresponding to between 1 and 2 g of nonvolatile matter (see (*g*)) to a 200 cc Erlenmeyer flask, reweigh the container and calculate the exact weight taken, add

25 cc of neutral pure benzol and 25 cc of neutral ethyl alcohol, or ethyl alcohol denatured with pure benzol or pure methyl alcohol. (The alcohol used in all cases must be neutral.) Boil for 30 minutes. (It is best to use a reflux condenser.) Cool to room temperature, add 4 drops of phenolphthalein indicator solution, and titrate with standard alcoholic sodium hydroxide. From the number of cubic centimeters of standard hydroxide solution calculate the acid number to the basis of the nonvolatile residue. (Acid number is milligrams of KOH required to neutralize acid in 1 g of material tested.)

4. REAGENT

Alcoholic Sodium Hydroxide Solution.—Dissolve pure sodium hydroxide in 95 per cent ethyl alcohol in the proportion of about 22 g per 1000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle and keep well stoppered. Standardize against half normal sulphuric or hydrochloric acid, using phenolphthalein as indicator. This standardization must be made each day that the solution is used.

This solution should be colorless or only slightly yellow when used, and it will keep colorless longer if the alcohol is previously treated with sodium hydroxide (about 80 g to 1000 cc) kept at about 50° C for 15 days and then distilled.

Washington, September 27, 1920.

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